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NATIONAL ADVISORY COMMITTEE FOR AERONAUTICS

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EFFECT OF ENVIRONMENTS OF SODIUM HYDROXIDE, AIR, AND ARGON ON THE STRESS-RUPTURE PROPERTIES OF NICKEL AT 1500° F

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SUMMARY

The stress-rupture properties of commercially pure nickel at 1500° F were determined for its application as a container material for molten sodium hydroxide. Stress-rupture tests of L-nickel tubes containing sodium hydroxide were run with atmospheres of air or argon external to the tubes. In addition, in order to establish the effects of oxidizing and inert environments on the strength of nickel tubes, various combinations of air and argon environments inside and outside of tubes devoid of sodium hydroxide were used.

Sodium hydroxide had little effect on the stress-rupture strength of nickel tubes tested in argon at 1500° F for times up to 200 hours. Mass transfer appeared to be the only form of corrosion taking place under these conditions.

Severe intergranular and surface oxidation increased the time-to-rupture of specimens wholly tested in air over that of specimens wholly tested in argon. This strengthening was accompanied by an embrittling effect of intergranular oxidation.

Strengthening by oxidation in air also occurred for tubes containing sodium hydroxide. This strengthening was less than that for tubes devoid of hydroxide, probably as a result of intergranular oxide forming in the tube walls to a sufficient depth for attack by the hydroxide.

Comparison of the times-to-rupture of nickel tubes and bars machined from the same bar stock and tested in air showed that the stress-rupture properties were dependent on the geometry of the specimen.

INTRODUCTION

Molten sodium hydroxide is of interest for nuclear reactors as a combination moderator-coolant. It has good heat-transfer characteristics, fair moderating ability, and good resistance to radiation damage (ref. 1). In addition, sodium hydroxide has a wide temperature range between its melting and boiling points (604° and 2534° F, respectively) which allows much freedom in the selection of operating temperatures.

These advantages are offset by the major problem of containing molten sodium hydroxide at elevated temperatures. Most materials are either readily dissolved or attacked intergranularly by sodium hydroxide at temperatures above 1000° F. The most promising corrosion-resistant container material is pure nickel. Even nickel is subject to a form of corrosion, commonly referred to as thermal-gradient mass transfer, in which metal is removed uniformly from the surface at a hot zone in the melt of sodium hydroxide and is deposited at a cooler surface. Reference 2 points out that, for temperatures up to 1500° F, mass transfer is the predominant form of corrosion taking place with unstressed nickel. No evidence of appreciable amounts of intergranular corrosion has been noted in unstressed nickel (refs. 3 and 4). In a reactor the material used as the container and piping for sodium hydroxide would be subjected to both mechanical and thermal stresses. In a stressed condition, the container might be weakened considerably by stress-induced acceleration of the corrosion. This investigation was conducted at the NACA Lewis laboratory to evaluate the strength of nickel at 1500° F in a sodium hydroxide environment and to determine whether the corrosion of nickel by sodium hydroxide at elevated temperatures would be accelerated, or the mechanism altered, by stress.

In addition, the gaseous environment that surrounds the piping system might have an effect on the strength of the piping (ref. 5). To explore the effect of an air environment, stress-rupture tests of nickel tubes containing sodium hydroxide were run in air, and the results were compared with data obtained in an inert environment (argon). Additional stress-rupture tests were run with the gaseous environment on both the inside and outside of the tube (no sodium hydroxide was used) in order to separate the effects of each environment and to obtain a better understanding of their interaction.

MATERIALS AND SPECIMENS

A nickel tube containing sodium hydroxide was selected for use in a stress-rupture test in order to determine the effect of sodium hydroxide on the strength of nickel. This type of specimen permitted the evaluation of the combined effects of sodium hydroxide and gaseous environments on the rupture strength of nickel.

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In order to evaluate the effects of specimen geometry on the rupture properties, nickel bars were also tested. The dimensions of the tube and bar specimens are shown in figure 1. These two types of specimen were obtained from different heats of L-nickel having the following chemical analyses and grain sizes:

Heat	Composition, percent by weight							Grain size, ASTM	
	Nickel	Carbon	Iron	Cobalt	Copper	Chromium	Silicon	As received	Annealed
Tube	99.23	0.030	0.36	0.43	0.06	0.004	0.003	7	4
Bar	99.30	.015	.15	.11	.09	.003	.006	4	2

In order to eliminate any effects due to differences in chemical composition and grain size, tube specimens were also made by drilling and machining tubes from the bar stock.

Most of the specimens were tested in the as-received condition. However, in order to evaluate the effects of recrystallization on the stress-rupture lives, several specimens were tested in air after annealing in a vacuum at 1900° F for 1 hour.

APPARATUS AND PROCEDURE

Stress-rupture tests were run at 1500° F on both bar and tube specimens. All bar specimens were tested in air. For tube specimens, different combinations of environment inside and outside the tube were used. The tests conducted are summarized in the following table:

Type of specimen	Heat	Heat treatment	Environment		Purpose
			External	Internal	
Tube	T	As received	Argon	Argon	Base line
	T		Argon	NaOH + argon[a]	Effect of NaOH
	T		Air	Argon	Effect of air, external
	T			Air	Effect of air, internal and external
	T			NaOH + argon[a]	Combined effect, air and NaOH
Bar	B			----------	Effect of geometry
Tube	B			Air	Eliminate chemistry effects
Tube	T	Annealed		Air	Effect of heat treatment on tubes
Bar	B	Annealed		----------	Effect of heat treatment on bars

[a]Argon was used as a cover gas over sodium hydroxide surface.

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Bar tests were conducted with standard stress-rupture equipment and procedure. For tube tests, the nickel tubes were welded to nickel grips as shown in figure 2. The upper grip was hollow and contained inlet and exit tubes for evacuating and for circulating gases inside the tube.

For stress-rupture tests of tubes in various environments, the following modifications were used:

(1) When air was the internal environment, the inlet and exit tubes in the upper grip were left open.

(2) For tests with an external environment of argon, an additional thin-walled tube was placed around the specimen (fig. 2), and argon was circulated in the annular area between the tubes.

(3) For tests with sodium hydroxide inside the tubes, a weighed amount of sodium hydroxide was placed in each specimen before welding on the lower grip. The assembly was then placed in the stress-rupture test furnace, and the water present in the hydroxide was removed by heating the specimen to approximately 700° F (above the melting point of sodium hydroxide) and simultaneously evacuating the tube to a pressure of 1 micron of mercury. The time required to reach this pressure was approximately 5 hours. A cleaned cover gas of argon was then introduced above the hydroxide melt, and a flow of about 2 liters per minute was continued during heating to the test temperature and throughout the time-to-rupture period.

The argon was purified by passing it through a column of molecular-sieve material (ref. 6) at room temperature to remove water vapor and, subsequently, through a bed of titanium chips at 1650° F to remove oxygen. A dew point of less than -100° F was attained with this purification train. In order to induce mass transfer during the stress-rupture test, the furnace temperature gradient was adjusted so that a temperature gradient of 40° F existed between the 1500° F hot section at the middle of the tube and the top of the sodium hydroxide melt.

Approximate creep measurements were made by following the movement of the grips of the stress-rupture machine with a dial gage. Elongation at rupture of the bar specimens was measured between gage marks placed on the shoulders of the specimen. Percentage elongation of these specimens was calculated by assuming that all the elongation took place within the 1-inch gage length.

The elongation at rupture of the tube specimens was measured over the total length of the tube. Since a temperature gradient existed along the tube, no gage length could be defined and, therefore, percentage elongation could not be calculated. In a few cases, the fracture of the tubes occurred in such a way that the two halves of the fractured

specimen could not be rejoined sufficiently to allow a meaningful measurement of the total elongation.

For tubes containing sodium hydroxide, the time for wall cracking, as indicated by a pressure drop in the argon cover gas, was recorded. The difference between this time and the time for complete tube rupture was less than 1 hour in all cases. For this reason, only the time to complete rupture was used for stress-rupture plots.

Stress-rupture tests at 1500° F were made on annealed (1900° F, 1 hr in a vacuum) and as-received tube and bar specimens to determine the effect of heat treatment. The results of these tests showed no difference in rupture strength due to the heat treatment. Metallographic examination revealed that recrystallization occurred in as-received specimens during the period of heating to test temperature in the stress-rupture machine (prior to loading). The structures and grain sizes that resulted from this recrystallization were similar to sizes produced in specimens annealed at 1900° F for 1 hour in a vacuum. For this reason, all subsequent specimens were tested in the as-received condition.

RESULTS AND DISCUSSION

A summary of the stress-rupture data is presented in table I and plotted in figures 3 and 4. The discussion is based on these strength data. Results of oxidation tests and microstructures will be discussed as required.

Effect of Sodium Hydroxide on Strength of Nickel in Argon.

A comparison of the stress-rupture curves for nickel tubes both containing and devoid of sodium hydroxide, in an argon atmosphere (fig. 3), shows that the hydroxide had no significant effect on the strength at 1500° F. These two curves appear to be coincident over the entire range of stress.

Microstructures of two tubes tested in argon at 1600 psi are shown in figure 5. The specimen on the left was tested with argon both inside and outside, while the specimen on the right contained sodium hydroxide on the inside and argon on the outside. In both specimens, intergranular cracks were formed in the grain boundaries perpendicular to the axis of stress. Although the photomicrographs show some difference in the density of intergranular cracking, this is not deemed to be particularly important. Of more importance is the fact that sodium hydroxide apparently has not attacked the nickel intergranularly.

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While the surface exposed to the hydroxide had metal removed by mass transfer, this removal was uniform and did not show preference for the grain boundaries. Figure 6 shows a specimen and the manner in which the nickel is deposited at the coldest portion of the tube. It was estimated that a nickel tube of the dimensions used in this study was reduced 8 percent in cross-sectional area in 200 hours exposure to sodium hydroxide with a difference in temperature of 40° F between the hot and cold sections of the tube. This amount of mass transfer is similar to that observed in unstressed nickel tubes (ref. 4).

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Strengthening Effect of Air

Comparison of the stress-rupture curves for tubes tested wholly in air (air inside and outside of the tubes) and tubes tested wholly in argon (fig. 3) shows that at 1500° F nickel tubes are much stronger in air than in argon. This strengthening effect of air becomes more pronounced with increasing time-to-rupture, so that the rupture curve for specimens tested in air tends to become horizontal. The creep rate of tubes tested in air for long lengths of time was observed to decrease progressively throughout the entire set of tests.

The rupture curve for tubes with air outside and argon inside is intermediate to those for tube tests conducted wholly in air or argon. For rupture times longer than 100 hours, the slope of this curve decreases and tends to become similar to that of the curve for tubes with air both inside and outside.

During stress rupture testing in air, the tubes became brittle. This embrittlement was so severe for tubes tested to rupture at stresses below 4000 psi (for long times) that a slight blow would fracture the tested specimens.

Intergranular cracks and voids appeared in all tube specimens tested wholly in argon below 5000 psi. Stress-rupture tests that were interrupted prior to failure showed that the cracks first started as small voids along those grain boundaries perpendicular to the axis of stress. With longer test times, the cracks grew and became continuous.

Tubes that were tested in air showed intergranular oxidation in the boundaries perpendicular to the axis of stress. Figure 7 shows the time dependence of the oxide growth in tubes tested in air at 3000 psi. These tests were interrupted at 139 hours and at 2000 hours (both prior to rupture) to obtain the specimens.

Intergranular oxidation varied from no visible intergranular oxide in unstressed nickel tubes in 50 hours at 1500° F to severe penetration of stressed specimens. Figure 8 shows the surface area of an unstressed

tube that was held at 1500° F for 50 hours and also a similar tube that was tested at 4000 psi for the same length of time. It is not known whether the intergranular oxide is formed as a result of grain boundary diffusion of oxygen, which takes place much more readily in stressed boundaries than in unstressed boundaries, or whether the oxygen enters the boundaries through intergranular cracks that are created during the stress-rupture testing and forms sufficient nickel oxide to fill the cracks.

All specimens tested above 5000 psi (having short rupture times) had ductile fractures with much elongation and necking, regardless of test atmosphere. The faster rate of deformation did not allow any intergranular cracking or oxidation. A comparison of the ductilities at fracture of tubes tested in various environments and at different stress levels is shown in figure 9.

The mechanism by which oxidation strengthens nickel is not completely understood. A surface layer of oxide reportedly can block the movement of dislocations and thus retard creep (refs. 7 to 9). High-temperature strengthening by oxidizing environments is also thought to result from internal effects (refs. 10 and 11). A major portion of the strengthening observed in this study is believed to result from the formation of nickel oxide in the grain boundaries. From the degree of intergranular oxidation occuring for long-time rupture tests (fig. 7), it is evident that the nickel oxide is capable of supporting much of the tensile load placed on the tube specimen.

Effect of Specimen Geometry on Strength of Nickel in Air

Figure 4 compares the stress-rupture curves for tubes and bars that were machined from the same stock of L-nickel and were tested in air. The curve for bar specimens is very nearly linear, while that for the tubes levels out with decreasing stress level (longer rupture times). As shown previously, this change in slope of the rupture curve for tubes can be explained by the strength contribution of the oxide which forms in long-time tests. Apparently oxidation strengthens the bars less than the tubes because of the lower surface-to-volume ratio of the bar. (Bar specimens had a surface-to-volume ratio of 16 sq in./cu in. compared with 30.6 sq in./cu in. for tubes tested with air inside and outside.)

In order to determine whether specimen geometry has any effect on rupture life other than that due to surface-to-volume ratio, a comparison of the rupture curves for tubes and bars having the same surface-to-volume ratio is of interest. Although data for material from the same heat of L-nickel are not available, an interesting comparison can be made between stress-rupture data for bars and for tubes (from tube stock) with air on the outside and argon inside. In this case, the ratio of surface

available for oxidation to volume is about the same (16 sq in./cu in. for the bar compared with 17.8 sq in./cu in. for the tube with air on the outside only). Since oxidation has been shown to strengthen nickel, it might be expected that tubes having air on one side would be stronger than the bars because the specimen thicknesses are different (1/4-inch diameter for the bar as compared with 1/16-inch wall thickness for the tubes). Thus, at a time when oxidation had progressed entirely through the tube wall, one-fourth of the bar area would still be unaffected. Contrary to this expectation, tubes with air only on the outside were considerably weaker than the bars for tests longer than 50 hours (compare figs. 3 and 4). This strength difference is apparently not attributable to differences in chemical composition or grain size, for comparison of the rupture curves for tubes from the different heats tested wholly in air indicates that the tube stock was slightly stronger. Response of both heats to oxidation was evidently similar, since the rupture curves for tubes from both heats tested wholly in air are approximately parallel. Apparently a specimen geometry effect, in addition to the surface-to-volume ratio, affects the rupture lives. In the tests just described, the unexpected weakness of the tubes with argon inside may be associated with the presence of an unoxidized internal surface that permits the relatively free excape of dislocations.

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Effect of Sodium Hydroxide on Strength of Nickel in Air

The rupture strength of tubes that contained sodium hydroxide and were tested in air is slightly less than the strength of tubes with air outside and argon inside for test times up to about 100 hours (fig. 3). For longer times, the curves become farther apart as the curve for tubes devoid of sodium hydroxide levels off. Since only a small portion of this difference results from mass transfer, the major difference is believed to result from the sodium hydroxide attack on the nickel oxide present in the grain boundaries. Removal of the oxide from these boundaries would limit the strengthening effect of oxidation. Strengthening probably occurs until the intergranular oxide penetration of the tube wall is sufficient for the sodium hydroxide to attack the oxide and cause failure. The progression of the oxide from the outside of the tube to the hydroxide side of the tube is shown in figure 10. This is an area very close to the fracture surface of the tube stressed at 2000 psi for 197 hours. Figure 11 shows the fracture surface of a specimen tested at 1500° F, for which failure occurred at 592 hours. The oxide is not present at the fracture surface, but it occurs in a few areas near the outside surface. The oxide coating on the outside of the tube near the fracture has been removed by the hydroxide which leaked out at the time of fracture. The oxide coating formed on the portion of the tube that did not come in contact with the hydroxide was several thousandths of an inch thick.

Although microstructural examination strongly indicated that rupture in air of tubes containing sodium hydroxide was associated with the hydroxide attack on nickel oxide in the grain boundaries, an alternate explanation of the weakening effect of sodium hydroxide is possible. Sodium hydroxide reacts with nickel to form sodium nickelite and hydrogen (refs. 12 and 13). If this hydrogen diffuses through the nickel lattice, it can reduce nickel oxide and thus limit the strengthening effect of oxidation. It is probable that both effects weaken the nickel tubes containing sodium hydroxide during stress-rupture testing in air.

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SUMMARY OF RESULTS

At 1500° F, the rupture lives of commercially pure nickel tubes are strongly dependent on the test environment inside and outside the tubes. The following effects of environment have been established:

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1. Nickel tubes have longer stress-rupture lives in air than in argon. This strengthening is accompanied by severe embrittlement from intergranular oxidation.

2. In an inert gas (argon), the stress-rupture lives of nickel tubes are not significantly affected by molten sodium hydroxide.

3. Sodium hydroxide reduces the stress-rupture lives of tubes tested in air, probably because of hydroxide attack of strengthening grain boundary oxides.

In the presence of a temperature gradient, unstressed nickel tubes containing sodium hydroxide are corroded primarily as a result of mass transfer. At 1500° F, the application of stress does not appear to increase the rate of mass transfer of nickel or to induce intergranular corrosion by sodium hydroxide.

CONCLUDING REMARKS

The strength of nickel at 1500° F has been shown to be strongly dependent on its environment. In addition, there is evidence that the response to environmental effects of relatively simple shapes such as bars and tubes may be different. It appears probable that the presence of unoxidized internal surfaces may weaken materials, possibly by providing a location for escape of dislocations. Further study is required to investigate this possibility.

Lewis Flight Propulsion Laboratory
National Advisory Committee for Aeronautics
Cleveland, Ohio, October 3, 1957

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TABLE I. - STRESS-RUPTURE DATA FOR DESCRIBED CONDITIONS OF ENVIRONMENT AT 1500° F[a]

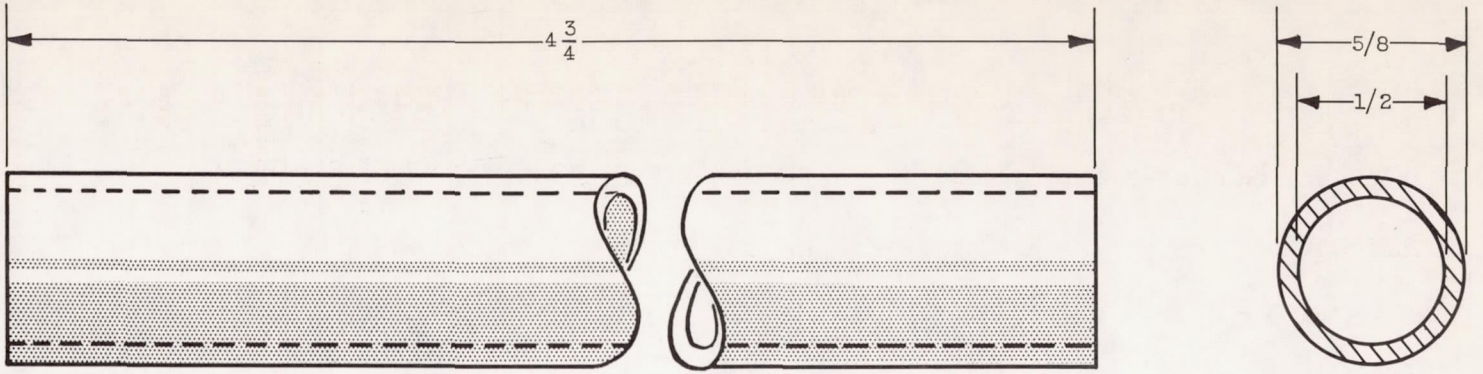
Stress, psi	Time to rupture, hr	Elongation, in.
Test condition (1)		
7000	1.0	1.56
6000	1.6	1.42
5500	3.7	1.26
4500	13.9	.57
4250	11.2	----
4000	17.0	.53
3250	36.9	.46
2500	43.6	.42
2250	74.2	----
2000	122.0	.42
1600	120.0	.22
1500	171.2	.24
Test condition (2)		
6500	1.3	1.31
5500	6.2	1.20
4000	15.0	----
3000	36.8	.48
3000	18.8	.33
2750	60.2	----
2250	55.7	----
1600	184.2	----
Test condition (3)		
5500	1.9	0.94
4240	20.2	.46
3865	22.6	.41
3000	67.6	.33
2750	70.2	.41
2000	367.1	.32
1700	1142.7	.22

Stress, psi	Time to rupture, hr	Elongation, in.
Test condition (4)		
6000	4.2	0.63
5000	9.9	.91
4590	17.3	.60
4500	28.0	----
4000	51.3	.41
3800	157.4	----
No fracture: 3500	2000	.47
No fracture: 3250	2000	.38
No fracture: 3000	2000	----
Test condition (5)		
6000	2.8	----
5000	11.0	0.76
4000	15.8	.55
3250	42.0	.33
3250	38.2	.36
3000	39.8	.39
2500	71.4	----
2000	197.4	.25
1500	592.2	.24
Test condition (6)		
7000	<0.5	115.0%
6000	1.5	108.7%
5000	4.4	90.6%
4000	30.0	-----
4000	24.3	86.0%
3500	78.0	84.0%
3000	680.9	59.5%
2750	846.7	31.8%
2500	1153.9	37.5%
Test condition (7)		
4500	5.4	0.49
4000	13.5	.42
3500	114.4	1.37
No fracture: 3250	1000.0	----

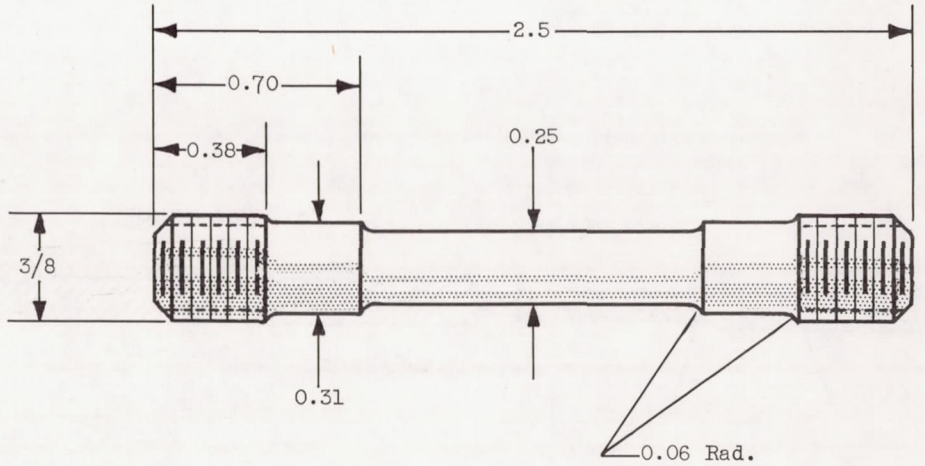
[a]Key to testing conditions:

Test condition	Geometry	Atmosphere		Heat of L - nickel (stock)	Heat treatment
		External	Internal		
1	Tube	Argon	Argon	Tube	As - received
2	↓	Argon	Argon + NaOH	↓	↓
3		Air	Argon		
4		Air	Air		
5		Air	Argon + NaOH		
6	Bar	Air	----------	Bar	
7	Tube	Air	Air	Bar	

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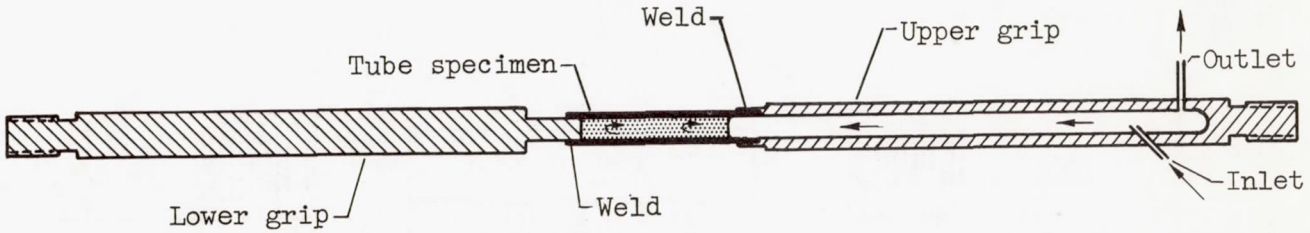
(a) Tube specimen.



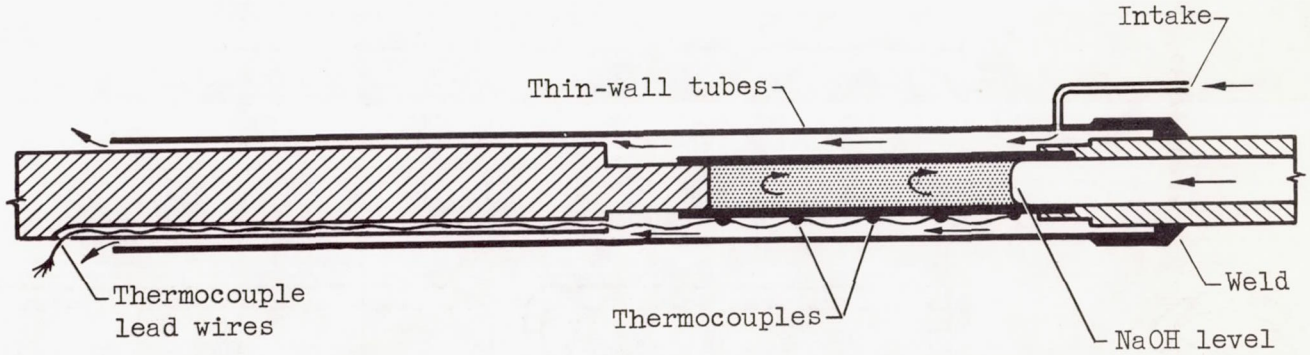
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(b) Bar specimen.

Figure 1. - Stress-rupture specimens. (All dimensions in inches.)



(a) For tests in air.



(b) For tests in argon.

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Figure 2. - Assembly of tube specimen and grips for stress-rupture testing.

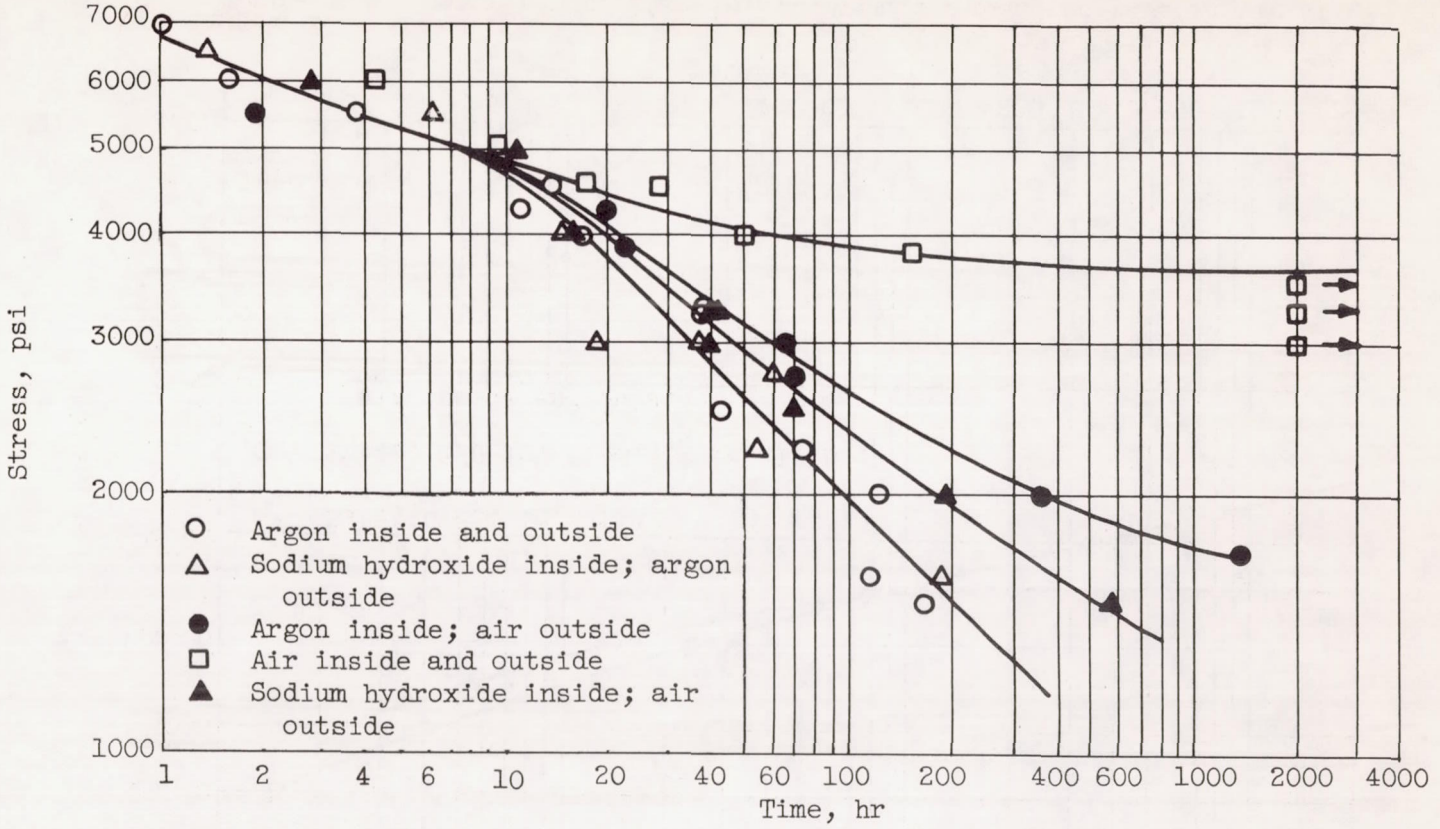


Figure 3. - Rupture life of tubes tested at 1500° F in various environments.

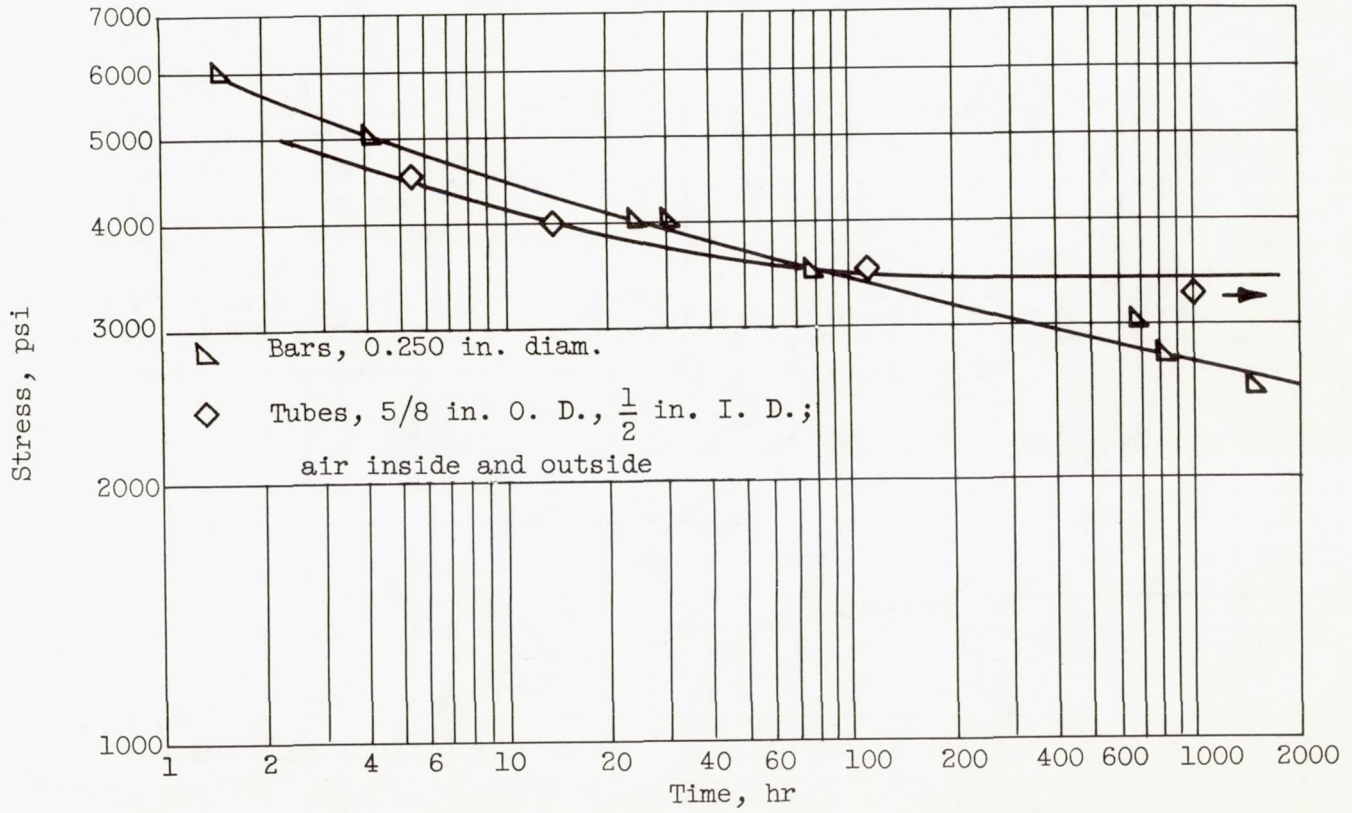
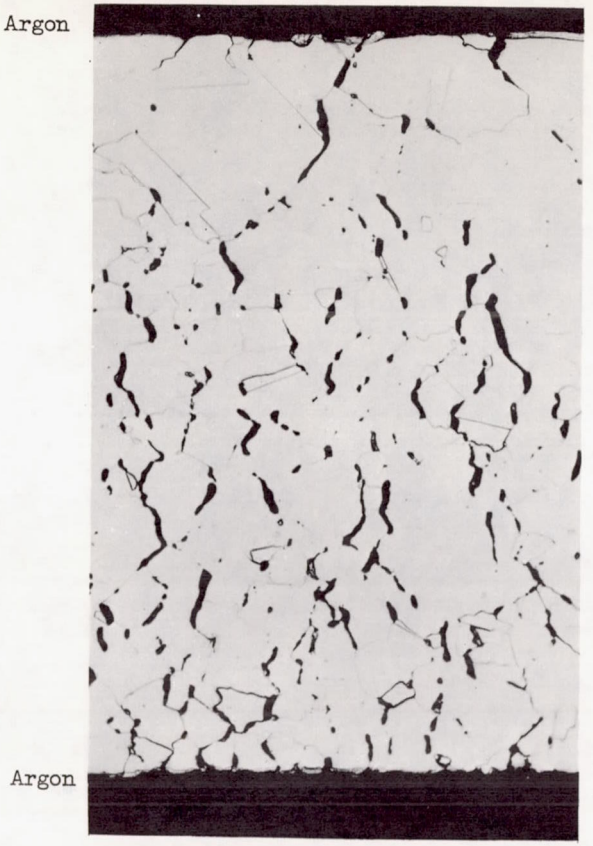
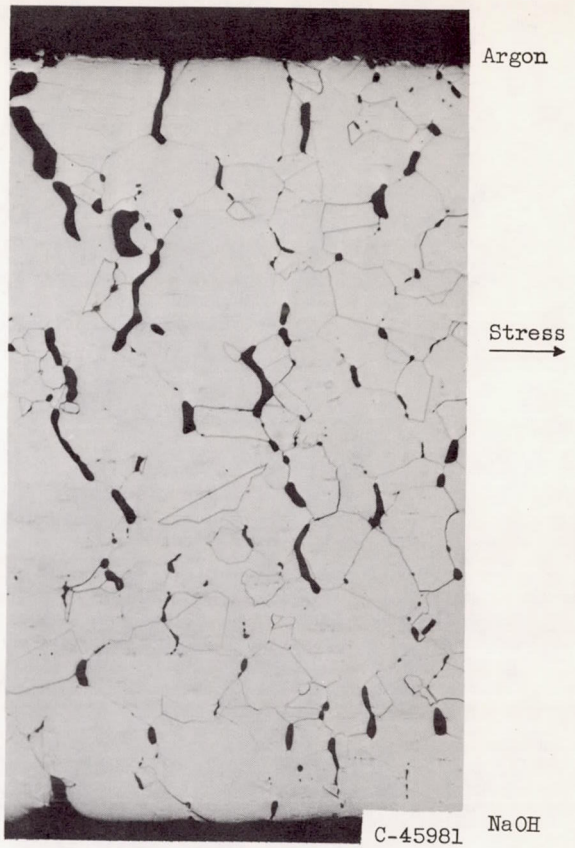


Figure 4. - Comparison of time-to-rupture for tubes and bars in air at 1500° F.



(a) Argon only; 120 hours.



(b) Argon outside, NaOH inside; 184 hours.

Figure 5. - Microstructure of tubes tested in argon at 1600 psi. Unetched. X75.

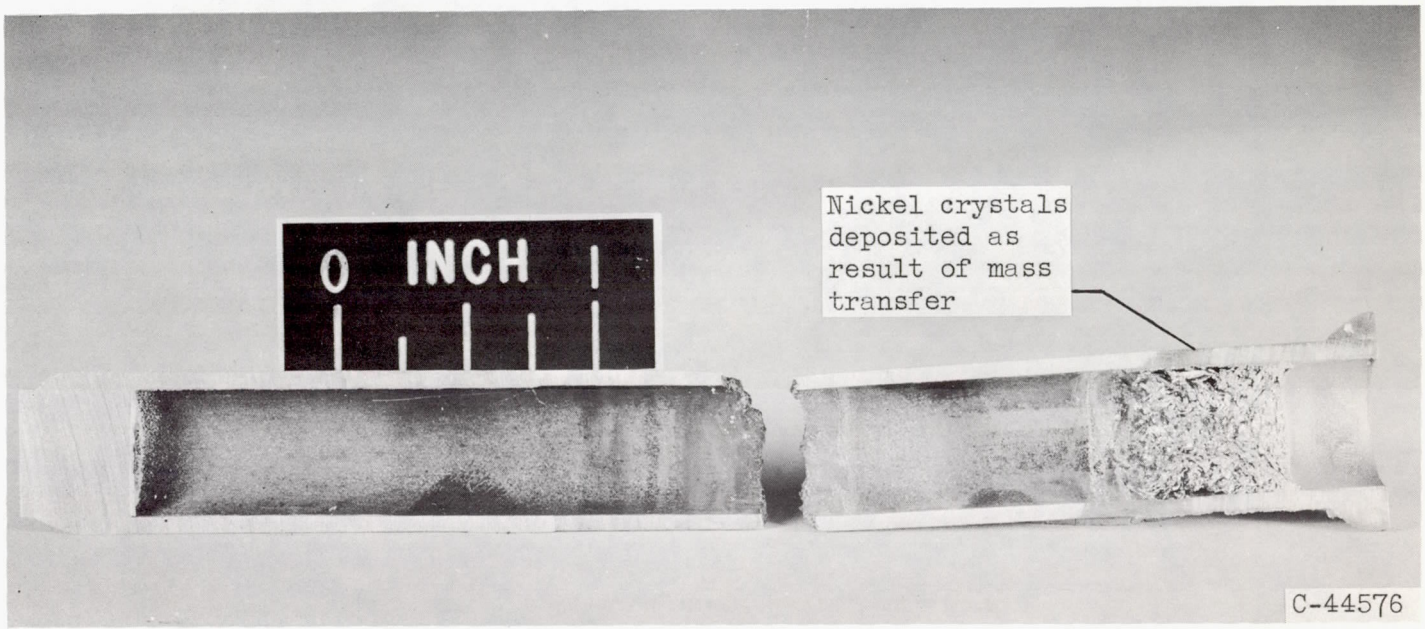
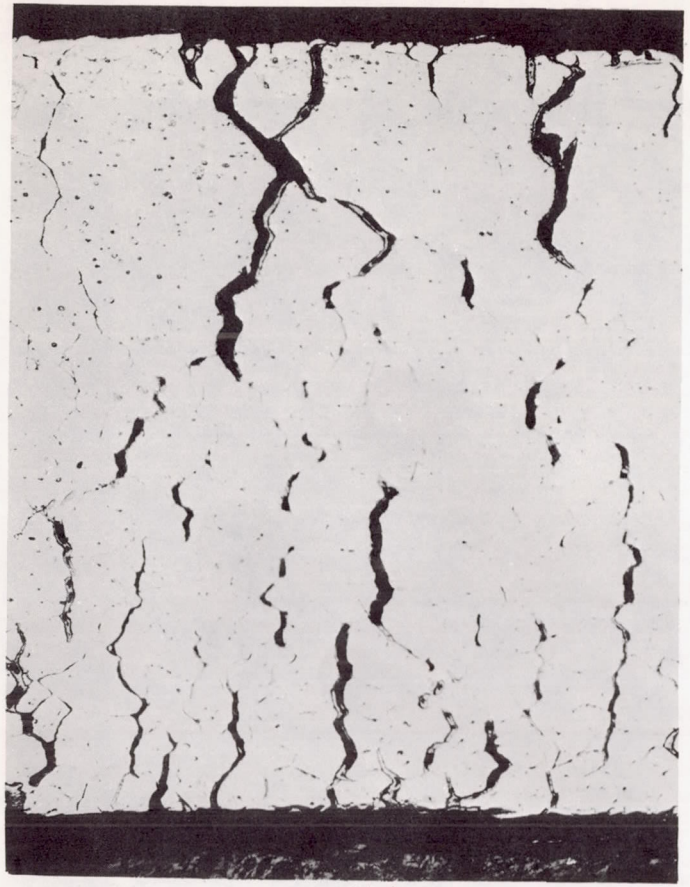


Figure 6. - Cross section of tube containing sodium hydroxide tested at 2000 psi for 200 hours.

Outside surface



(a) 139 Hours.

Outside surface



(b) 2000 Hours.

Figure 7. - Effect of time on intergranular oxidation at 3000 psi and 1500° F. (Specimen had not fractured in 2000 hr.) Unetched. X75.

(a) No stress.

(b) 4000 psi.

Figure 8. - Effect of stress on rate of intergranular oxidation at 1500° F. Unetched; 50 hours. X75.

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Test Conditions

Tube	Atmosphere	Stress, psi	Time, hr.
1	Argon inside and outside	6000	1.6
2	Argon inside and outside	3000	35.2
3	NaOH inside; argon outside	3000	36.8
4	Air inside and outside	5000	9.9
5	Argon inside; air outside	1700	1142.7
6	NaOH inside; air outside	2000	197.4

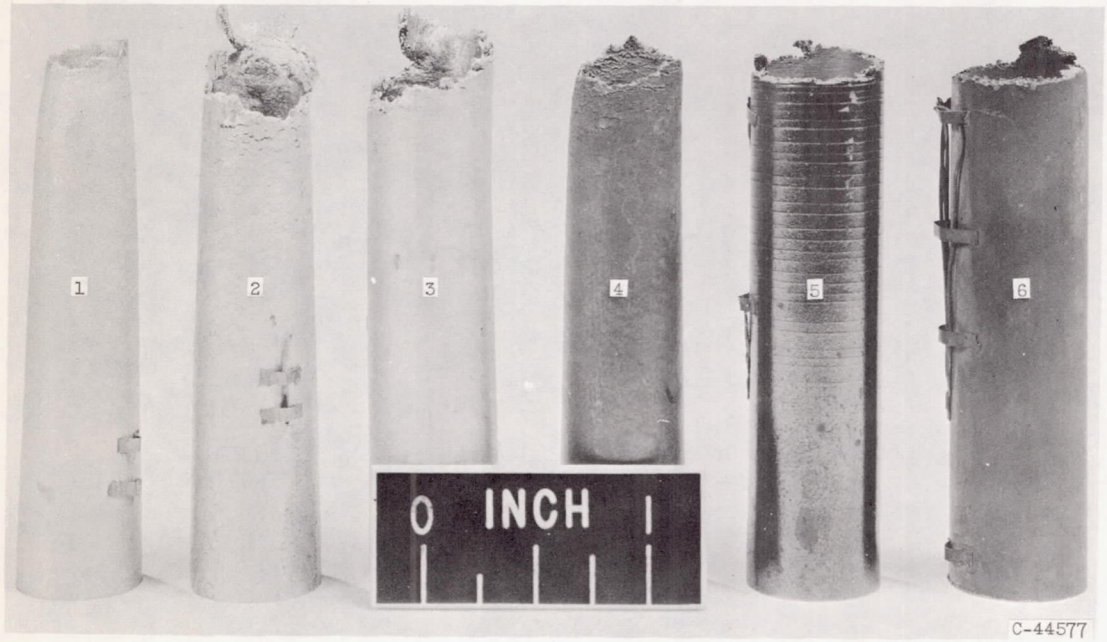


Figure 9. - Fracture edges of tubes for various test conditions of stress and environment.

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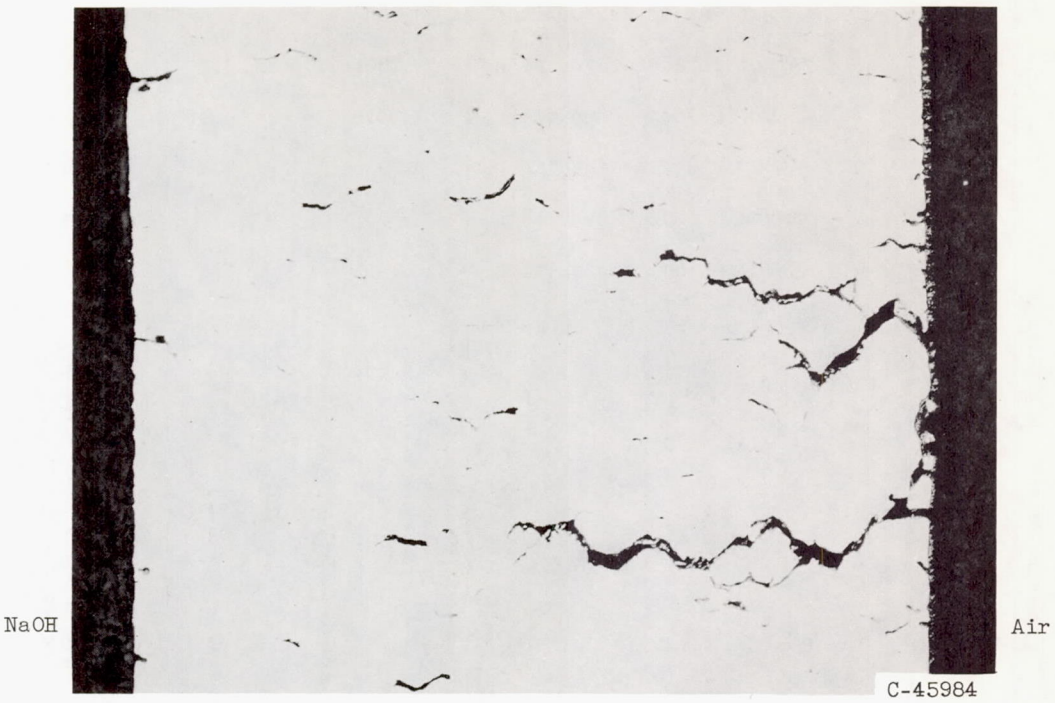


Figure 10. - Intergranular oxide penetration of tube that failed in 197 hours at 2000 psi and 1500° F. Unetched. X75.

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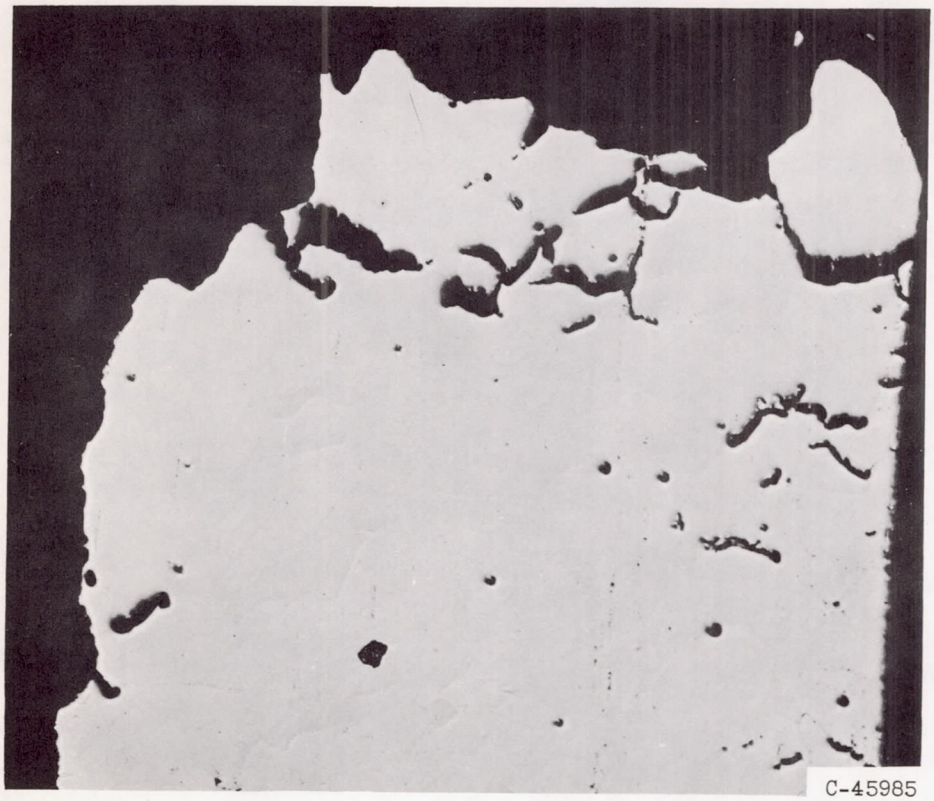


Figure 11. - Fracture of tube that contained NaOH and was tested in air at 1500 psi and 1500° F for 592 hours. Unetched. X75.